

Improvements in or relating to tobacco smoke-filtering elements

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Abstract of **GB 896599 (A)**

Filtering elements for cigarettes, tobacco pipes or cigar or cigarette holders comprise a bundle of continuous synthetic filaments with particles of a water-insoluble calcium or magnesium salt distributed over their surfaces and bonded thereto by an agent miscible with, or a solvent for, the filament material. The filaments may be bonded together at random points. A salt of meta-, ortho- or pyro-phosphoric acid or of stearic or carbonic acid may be used, and the particles may be between 0.1 and 10 microns in size and form from 5% to 60% of the filtering element. The major portion of the particles may be of a size less than twice the filament diameter but greater than one-tenth the filament diameter, and preferably, at least 80% of the particles are of less than filament diameter. The particles may be blown on to the filaments, or applied thereto as a slurry in cold water or in a volatile organic vehicle, or they may be applied electrostatically to filaments in the form of a banded tow. Alternatively, the particles may be dusted on to a banded tow of filaments, the surface of which is wetted with adhesive, e.g. dextrin glue, or with plasticizer, before, during or after dusting, e.g. a slurry of plasticizer, salt particles and perhaps water or alcohol may be used. The filaments may be nylon, polyamide, polyester or regenerated cellulose, or of cellulose acetate having 4 to 10 crimps per inch, 3 to 16 denier and 4,000 to 35,000 filaments to a bundle. The plasticizer may be glycerol triacetate or tripropionate, triethyl citrate, or di-(methoxyethyl) phthalate with or without sodium lauryl sulphate. After treatment with plasticizer and particles, a banded tow may be returned to its original shape and bonded by heat treatment after forming into paper-wrapped cigarette filter rod lengths. To aid particle bonding, the filament surfaces may be treated with an oil, before or after adding the plasticizer and particles. The oil may comprise from 3% to 8% of the filtering element and is colourless, odourless, tasteless, non-volatile and non-toxic, and insoluble in the plasticizer at room temperatures although it may be soluble therein at elevated temperatures so that it may be applied dissolved in the plasticizer applied hot. Suitable oils are castor, cottonseed, almond, coconut, peanut and olive; pharmaceutical grades of light, medium and high viscosity mineral oils, lauryl alcohol and glycerol oleates, laurates and ricinoleates. Quantitative process examples are given.

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PATENT SPECIFICATION

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COMPLETE SPECIFICATION.

Improvements in or relating to Tobacco Smoke-Filtering Elements.

- We, EASTMAN KODAK COMPANY, a Company organized under the laws of the State of New Jersey, United States of America, 343 State Street, Rochester, New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—
- The present invention relates to tobacco smoke filtering elements suitable for use in cigarettes, tobacco pipes, cigarette holders, and cigar holders.
- In accordance with the present invention there is provided a tobacco smoke filtering element comprising a bundle of continuous synthetic filaments having finely divided particles of a water-insoluble calcium or magnesium salt distributed over the surfaces of the filaments and bonded thereto by means of a bonding agent compatible with the material of the filaments. By compatible with the material of the filaments we mean that the bonding agent comprises a substance which is miscible with or is a solvent for that material. Preferably, the salt is the salt of an acid which is weaker than acetic acid.
- In carrying the invention into effect, the filaments of a pervious fibrous filter mass are made to carry a dispersed deposit of a finely divided, solid, inorganic salt consisting of the phosphates or carbonates of magnesium or calcium. In general, the spacing of the filaments provides minute passages for smoke through the mass, the mass carrying therein an additive comprising finely divided solid particles of a water-in-
- soluble inorganic salt of the type described above in the amount of about 5% to 60%, preferably 5% to 30%, by weight of the mass plus additive, the concentration not being critical so long as there is sufficient additive present to improve the filtration efficiency of the filter without creating excessive pressure drop, said particles being supported along the surfaces of and by the filaments and occupying positions along the walls of and partially blocking said passages whereby a substantial portion of the smoke is impinged on the particles.
- Any suitable means known to the art for spreading a powder on to a fibrous surface or through a fibrous mass may be employed in spreading filters of the invention. Thus, for example, the inorganic salt powder can be blown on to the filaments or it can be applied as a slurry in cold water or in a volatile organic vehicle. Another method is to apply the powder to the filaments electrostatically, i.e. to induce a charge on the filaments by friction or other suitable means and then to run the filaments through a chamber containing a highly concentrated cloud of salt dust. Preferably this is done with the filaments in the form of a tow and in a banded, i.e. spread-out, condition. Still another method for applying the salt particles is to wet the surface of the filaments with an adhesive or a plasticizer before exposing them to a solids dusting device or other solids applicator. That is to say, tow from a supply roll is opened to debundlize the filaments and provide a larger and more uniform tow cross-section, and the opened tow is spread uniformly to a much larger width of e.g., 10 times its

[Price 4s. 6d.]

original width, thereby exposing substantially all of the filaments to material, i.e. plasticizer, issuing from a dispenser adjacent which the tow passes. The dust may be added before, simultaneously with, or after the plasticizer, preferably with or after, and most advantageously with the plasticizer. In the latter case the plasticizer first is converted to a slurry containing the finely divided inorganic salt and, if necessary, a diluent such as water or alcohol. The diluent must be a non-solvent for the powder but may dissolve in or emulsify with the plasticizer. We have found that finely divided solids which have a tendency to sift out of fibrous filter masses generally are more firmly bound if applied in a plasticizer slurry. Also, by use of a slurry for application of the solid it has been possible to load the filaments with larger concentrations of uniformly dispersed and firmly bound additive. Employment of a diluent inert to the powder and to the filaments is valuable in facilitating application of larger quantities of additive without a corresponding increase in the quantity of plasticizer. The invention is further illustrated in the following examples:—

EXAMPLE 1.

Magnesium Carbonate.

A partially opened tow of a crimped, regular cross section cellulose acetate yarn (39.2% acetyl) having about eight crimps per inch and containing approximately 10,000 filaments of 8 denier per filament was slowly pulled over a compressed air banding device which spread out the filaments to a width of about twelve inches. While the tow was in this spread condition it was sprayed (using a conventional point spray gun) uniformly with finely divided droplets of di(methoxyethyl) phthalate plasticizer in order to make numerous portions of the surfaces of the filaments soft and tacky. The spread tow was then passed through a dusting chamber which dusted the fibers uniformly with a finely divided (No. 325, U.S. Sieve Series) powder of magnesium carbonate ($MgCO_3$) of a type commercially available and designated as analytical grade. On leaving the dusting chamber the tow contained 14% plasticizer and 18% mag-

nesium carbonate, based on the total weight of tow, carbonate and plasticizer. After this spreading, spraying, and dusting, the treated tow was pulled back to its original shape of a compact bundle, i.e. cord, and fed into a conventional cigarette filter plug making machine which formed it cylindrically, wrapped it with paper, and cut it into rods 78 mm. in length and 7.5 mm. in diameter. The rods were then given a heat treatment for one hour at 80° C. to cause partial solvation of the acetate filaments by the plasticizer and thereby bond the carbonate particles to the filaments and the filaments to each other, imparting firmness to the rods. Thereupon the rods were readily cut into 13 mm. filter tip lengths and attached to kingsize (85 mm.) cigarettes of a standard brand available on the retail market in the U.S. The cigarettes were first shortened by 13 mm. and the 13 mm. rods then affixed to the shortened cigarettes by means of an adhesive tape. These filter cigarettes were smoked to a total butt length of 35 mm. on a smoking machine similar in design and operation to the smoking machine described by J. A. Bradford, W. R. Harlan and H. R. Hanmer in *Industrial and Engineering Chemistry*, Vol. 28, pp. 836-9 (1936). The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content.

Control filters without a powdered additive were also prepared from the same cellulose acetate tow containing the same amount of di(methoxyethyl) phthalate plasticizer. These filters were attached to the same brand of standard king size cigarettes shortened by 13 mm. These cigarettes were also smoked to a total butt length of 35 mm. by means of the smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content.

Ten unfiltered king size cigarettes (same brand) which were not shortened by 13 mm. were smoked to a butt length of 35 mm. by means of the smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content.

The results obtained from the three sets of cigarettes are listed in Table I.

TABLE I.

	Average Pressure Drop*	mg. Tars Found in The Smoke	mg. Nicotine Found in The Smoke
Cigarettes without filters (85 mm. length)	3.5"	163	25
Cigarettes with cellulose acetate filters	4.0"	148	23
Cigarettes with cellulose acetate—magnesium carbonate filters	4.2"	92	14

*Pressure drop in inches of water at an air flow rate of 17.5 cc/sec through the cigarette.

From the results listed above it can be seen that the filter containing the combination of acetate filaments and magnesium carbonate reduced the amount of tar collected by 43.6% and the amount of nicotine collected by 44%. The control filter reduced the amount of tars collected by 9.2% and the amount of nicotine collected by 8%. The increase in pressure drop of the filtered cigarette due to the addition of the carbonate to the filter was only about 5%.

EXAMPLE 2.

Calcium Carbonate.

The procedure described in Example 1 was repeated using the same tow of cellulose acetate and the same plasticizer but using in place of the magnesium carbonate a powdered calcium carbonate (CaCO_3) capable of passing through a No. 325 screen (U.S. Sieve Series). The treated tow contained 14% plasticizer and 20% calcium carbonate (based on the total weight of the treated tow) as it left the dusting chamber. The baked plugs were cut into 13 mm. filter tips and attached to the standard king size cigarettes which had been shortened by 13 mm. These filtered cigarettes were smoked to a total butt length of 35 mm. on the smoking machine operated under the same conditions as in Example 1, and the smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content.

The collected smoke contained 90 milligrams of tar and 13 milligrams of nicotine. This corresponds to a reduction in tar of 44.6% and a reduction in nicotine of 48.0% due to the filter. The cigarettes had an average pressure drop of 4.3 inches of water.

EXAMPLE 3.

Calcium Carbonate.

The procedures described in Example 1 were repeated using a tow of cellulose acetate yarn containing 5,000 filaments of 16 denier per filament, a plasticizer consisting of triethyl citrate, and a powder consisting of calcium carbonate (CaCO_3) with an average particle size of 0.1 micron. The treated tow contained 60% cellulose acetate, 30% calcium carbonate, and 10% plasticizer. A control tow of cellulose acetate yarn of 5,000 filaments, 16 denier per filament, and containing 14% triethyl citrate, was also prepared. Both treated tow samples were processed into the filter rods and the rods were heated at 60° C. for one hour. Filter tips of 13 mm. in length were prepared from both sets of rods and placed on the standard king size cigarettes which had been shortened by 13 mm. Ten cigarettes containing the calcium carbonate filters and 10 cigarettes containing the control filters were smoked to butt lengths of 35 mm. on the smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content.

	Average Pressure Drop	mg. Tars Found in The Smoke	mg. Nicotine Found in The Smoke
Cigarettes with cellulose acetate filter (85 mm. length)	3.8"	155	24
Cigarettes with cellulose acetate- CaCO_3 (85 mm. length)	4.1"	115	18

From the values listed above and in Example 1 it can be seen that the filter containing the combination of acetate filaments and calcium carbonate reduced the amount of tars collected by 29.4% and the amount of nicotine collected by 28.0%. The control filter reduced the amount of tars collected by 4.9% and the amount of nicotine collected by 4.0%. The increase in pressure drop of the filtered cigarette due to the addition of the calcium carbonate to the filter was only about 5%.

EXAMPLE 4.

Calcium Carbonate.

A tow of regenerated cellulose yarn (viscose) containing 16,000 filaments of 6 denier per filament was spread out to a width of 10 inches and sprayed with a 25% aqueous solution of dextrin glue. While the

tow was still spread out and wet with the glue it was passed through a dusting chamber where it was uniformly covered with a fine calcium carbonate dust of 0.1 micron particle size. On leaving the dusting chamber the tow consisted of 40 parts regenerated cellulose filaments, 30 parts glue (25% solution) and 30 parts calcium carbonate. The combination was pulled back into the form of a compact bundle or cord and fed into a filter-rod making machine which paper wrapped it and cut it into filter plugs 78 mm. in length and 7.5 mm. in diameter. The rods were heated for 1 hour at 100° C. to dry the glue and bond the powder to the filaments. The plugs were then cut into 13 mm. filter tip lengths and attached to 85 mm. standard king size cigarettes which had been shortened by 13 mm. Ten of these cigarettes were smoked to a total butt

length of 35 mm. on the smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content.

- 5 A control tow sample, consisting of 16,000 filaments of the 6 denier per filament regenerated cellulose and containing 43% of dextrin glue (25% solution) but no calcium carbonate, was prepared in a similar
10 manner. The plugs from this tow were

heated for 1 hour at 100° C. and cut into filter tips 13 mm. in length. These tips were attached to 85 mm. standard king size cigarettes which had been shortened by 13 mm. Ten of these cigarettes were
15 smoked to a total butt length of 35 mm. on the smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content.

	Average Pressure Drop	mg. Tars Found in The Smoke	mg. Nicotine Found in The Smoke
20 Cigarettes with the regenerated filter (85 mm. length)	4.1"	140	21
25 Cigarettes with the regenerated cellulose-CaCO ₃ filter (85 mm. length)	4.3"	100	16

- 30 From the values listed above and in Example 1 it can be seen that the filter containing the combination of regenerated cellulose filaments and calcium carbonate reduced the amount of tars collected by 38.7% and the amount of nicotine collected by 36.0%. The control filter of regenerated cellulose filaments reduced the amount of
35 tars collected by 14.1% and the amount of nicotine collected by 16.0%. The increase in the pressure drop of the filtered cigarette due to the addition of the calcium carbonate to the filter was only about 5%.

EXAMPLE 5.

Calcium Carbonate.

- 40 A tow of cellulose acetate yarn containing 12,000 filaments of 5 denier per filament was spread to a width of 12 inches and sprayed with an emulsion consisting
45 (by weight) of 0.03 part sodium lauryl sulphate, 1 part di-(methoxyethyl)phthalate plasticizer, 2 parts water, and 3 parts calcium carbonate powder of ultimate particle size of approximately 6 microns. The
50 spread tow was then pulled back into a compact bundle or cord and fed into a cigarette filter plug making machine which paper wrapped it and cut it into filter rods 78 mm. in length and 8 mm. in diameter. The rods
55 were heated for thirty minutes at 100° C. to evaporate the water and to bond the calcium carbonate firmly to the filaments by means of the plasticizer. Analysis of the
60 plugs indicated that they consisted of 51% cellulose acetate filaments, 33% calcium carbonate, 5% moisture, and 11% di-(methoxyethyl) phthalate plasticizer. The heated and cooled plugs were quite firm
65 and the calcium carbonate was firmly

bonded to the filaments. The plugs were cut into 13 mm. filter tip lengths and attached to standard American king size (85 mm.) cigarettes which had been shortened by 13 mm. These filtered cigarettes were smoked to a total butt length of 35 mm. on the standard smoking machine described in Example 1. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content.
70 75

A tow of cellulose acetate yarn containing 20,000 filaments of 5 denier per filament was spread to a width of 12 inches and sprayed with an emulsion consisting of 0.03 part sodium lauryl sulphate, 1 part di-(methoxyethyl) phthalate plasticizer, and 2 parts water. This tow, containing no calcium carbonate, was converted into filter plugs in the manner described above and the plugs were dried for thirty minutes at 100° C. Analyses of these plugs indicated that they consisted of 84% cellulose acetate filaments, 12% di-(methoxyethyl) phthalate plasticizer, and 4% moisture. The plugs were cut into 13 mm. filter tips and attached to ten standard king size cigarettes shortened by 13 mm. These cigarettes were also smoked to total butt lengths of 35 mm. on the smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content.
80 85 90 95

Ten unfiltered, standard king size cigarettes which were not shortened by 13 mm. were smoked to a butt length of 35 mm. by means of the smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content.

The results obtained from the three sets of cigarettes are listed in Table II.
100 105

TABLE II.

		Average Pressure Drop*	mg. Tars Found in The Smoke	mg. Nicotine Found in The Smoke
5	Unfiltered cigarettes (85 mm.)	3.3"	165	25
	Cigarettes, 72 mm. in length, containing 13 mm. acetate filter of 20,000 filaments	4.2"	130	20
	Cigarettes, 72 mm. in length, containing 13 mm. acetate-CaCO ₃ filter	4.2"	93	13

10 *Pressure drop in inches of water at an air flow rate of 17.5 cc./second through the cigarette.

From the results listed above, it can be seen that the filter containing the combination of cellulose acetate filaments and calcium carbonate reduced the amount of tars collected by 44% and the amount of nicotine collected by 48%. The filter containing the larger number of cellulose acetate filaments but no calcium carbonate reduced the amount of tars by only 21% and the amount of nicotine by 20%, although its average pressure drop was equal to that of the cellulose acetate-calcium carbonate filter.

EXAMPLE 6.

Calcium Carbonate.

A tow of cellulose acetate yarn containing 14,000 filaments of 5 denier per filament was spread to a width of 12 inches and sprayed with a suspension of 51% calcium carbonate powder in di(methoxyethyl) phthalate plasticizer containing 33% ethyl alcohol. The calcium carbonate powder had an ultimate particle size of 3 microns.

The spread tow was pulled back into a compact bundle or cord and fed into a cigarette filter plug making machine which paper wrapped it and cut it into filter rods 78 mm. in length and 8 mm. in diameter. The rods were heated for thirty minutes at 80° C. to evaporate the ethyl alcohol and bond the calcium carbonate powder to the filaments by means of the plasticizer. Analyses of the plugs indicated that they consisted of 65% cellulose acetate filaments, 20% calcium carbonate powder, and 13% plasticizer. The heated and cooled plugs were quite firm and the calcium carbonate was firmly bonded to the filaments. The plugs were cut into 13 mm. filter tips and attached to standard American king size cigarettes which had been shortened by 13 mm. These filtered cigarettes were smoked to a total butt length of 35 mm. on the standard smoking machine described in Example 1. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content.

The collected smoke contained 111 mg. of tar and 15 mg. of nicotine. This corresponds to a reduction in tar of 33% and a reduction in nicotine of 40% due to the filter when the values obtained are com-

pared with the unfiltered cigarette values listed in Example 5. The filtered cigarettes had an average pressure drop of 4.3 inches of water.

EXAMPLE 7.

Calcium Carbonate.

A tow of cellulose acetate yarn containing 14,000 filaments of 5 denier per filament was spread to a width of 12 inches and sprayed with a suspension of 58% calcium carbonate powder in glycerol tripropionate plasticizer. The calcium carbonate powder had an ultimate particle size between 7 and 10 microns.

The spread tow was pulled back into a compact bundle or cord and fed into a cigarette filter plug making machine which paper wrapped it and cut it into filter rods 78 mm. in length and 8 mm. in diameter. Within one hour at room temperature the rods were rigid and the calcium carbonate was firmly bonded to the filaments. The plugs were cut into 13 mm. filter tips and attached to standard king size cigarettes which had been shortened by 13 mm. These filtered cigarettes were smoked to a total butt length of 35 mm. on the standard smoking machine described in Example 1. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content.

The collected smoke contained 108 mg. of tar and 15 mg. of nicotine. This corresponds to a reduction in tar of 35% and a reduction in nicotine of 40% due to the filter when the values are compared with the unfiltered cigarette values listed in Example 5. The filtered cigarettes had an average pressure drop of 4.2 inches of water.

EXAMPLE 8.

Magnesium Phosphate Mg₃(PO₄)₂

A crimped tow of the type employed in Example 1 was treated as described in that example with magnesium phosphate (No. 325, U.S. Sieve Series) rather than magnesium carbonate as employed in Example 1. On leaving the dusting chamber, the tow of filaments contained 14% plasticizer and 18% magnesium phosphate based on the total weight of the combination. Filters were made from this treated tow and

fastened to cigarettes which were then test smoked, all by methods as described in Example 1. Analytical data obtained showed that cigarettes with cellulose acetate-magnesium phosphate filters (85 mm. length) had an average pressure drop of 4.2 inches. The smoke contained 98 mg. of tars and 15 mg. nicotine. Comparison of this data with the data obtained for controlled filters and unfiltered cigarettes in Example 1 shows that the magnesium phosphate filter reduced the amount of tars collected by 40.0% and the amount of nicotine by 40.0%. The increase in pressure drop of the filtered cigarettes due to the addition of the magnesium phosphate to the filament was only about 5%.

EXAMPLE 9.

Calcium Phosphate $\text{Ca}_3(\text{PO}_4)_2$

The procedure described in Example 1 was repeated using the same tow of cellulose acetate and the same plasticizer but using a powdered calcium phosphate $\text{Ca}_3(\text{PO}_4)_2$ capable of passing through a No. 325 screen (U.S. Sieve Series) in the dusting chamber. The tow of filaments prepared in this case contained 14% plasticizer and 20% calcium phosphate (based on the total weight) as it left the dusting chamber. The baked plugs were cut into 13 mm. filter tips and attached to the standard king size cigarettes which had been shortened by 13 mm. These filtered cigarettes were smoked to a total butt length of 35 mm. on the smoking machine and the smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content.

The collected smoke contained 100 milligrams of tar and 16 milligrams of nicotine. This corresponds to a reduction in tar of 38.6% and a reduction in nicotine of 36.1% due to the filter. The cigarettes had an average pressure drop of 4.4 inches of water.

EXAMPLE 10.

Calcium Pyrophosphate. $\text{Ca}_2\text{P}_2\text{O}_7$

The procedure described in Example 1 was repeated using a tow of cellulose acetate yarn containing 5,000 filaments of 16 denier per filament, a plasticizer consisting of triethyl citrate, and a powder consisting of calcium pyrophosphate, $\text{Ca}_2\text{P}_2\text{O}_7$, capable of passing through a No. 325 screen (U.S. Sieve Series). The treated tow contained 60% cellulose acetate, 30% calcium salt, and 10% plasticizer. A control tow of cellulose acetate yarn of 5,000 filaments, 16 denier per filament, and containing 14% triethyl citrate, was also prepared. Both treated tow samples were processed into filter rods and the rods were heated at 60° C. for one hour. Filter tips of 13 mm. in length were prepared from both sets of rods and placed on the standard king size cigarettes which had been shortened by 13 mm. Ten cigarettes containing the calcium pyrophosphate filters and 10 cigarettes containing the control filters were smoked to butt lengths of 35 mm. on the smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content. Results were as shown in Table III.

TABLE III.

	Average Pressure Drop	mg. Tars Found in The Smoke	mg. Nicotine Found in The Smoke
Cigarettes with cellulose acetate filter (85 mm. length)	3.8"	155	24
Cigarettes with cellulose acetate- $\text{Ca}_2\text{P}_2\text{O}_7$ filter (85 mm. length)	4.2"	110	17

From the values listed above and in Example 1, it can be seen that the filter containing the combination of acetate filaments and calcium pyrophosphate reduced the amount of tars collected by 32.6% and the amount of nicotine collected by 32.0%. The control filters reduced the amount of tars collected by 4.9% and the amount of nicotine collected by 4.0%. The increase in pressure drop of the filtered cigarette due to the addition of the calcium pyrophosphate to the filter was only about 5%.

EXAMPLE 11.

Calcium Diphosphate. CaHPO_4

A tow as described in Example 4 was

treated and converted into filters as described in that example, except that calcium diphosphate (capable of passing through a No. 325 screen, U.S. Sieve Series), was used in place of the calcium carbonate. Rods were made and tested in the same way. They contained 45 parts regenerated cellulose filaments, 30 parts glue (25% solution) and 30 parts CaHPO_4 powder. The filters were evaluated by test smoking as conducted in Example 4 and comparison to control filters and unfiltered cigarettes as tested in that example. The filters containing the calcium acid phosphate had an average pressure drop of 4.3 inches and their smoke

contained 102 mg. tar and 16 mg. nicotine.

From the values listed above and in Example 1, it can be seen that the filter containing the combination of regenerated cellulose filaments and calcium diphosphate reduced the amount of tars collected by 37.4% and the amount of nicotine collected by 36.0%. The control filter of regenerated cellulose filaments reduced the amount of tars collected by 14.1% and the amount of nicotine collected by 16.0%. The increase in the pressure drop of the filtered cigarette due to the addition of the calcium acid phosphate to the filter was only about 5%.

Under some conditions, particularly with rough handling there may be a tendency in some instances for some of the additive to shift out of the filters. This tendency may be obviated by adding a water-insoluble, oily material to the surface of the filaments which are to be used in making a filter. Preferably this oily addition is made prior to the application of the powdered additive. The choice of the particular oily material to spray on the filaments is of some importance. The oil should be insoluble at room temperatures in the plasticizer which is also applied to the filaments. The reason for this is that the oil will then remain on the surface of the filaments rather than be absorbed into the filaments as in the case of the plasticizer. The oil should be substantially colourless, odourless, tasteless, non-volatile and nontoxic. Although insolubility of the oil in the plasticizer at room temperatures is an essential property, the oil can be soluble in the plasticizer at elevated temperatures. This offers the possibility of dissolving the oil in hot plasticizer solutions and spraying it on the spread-out filaments in a one-step operation. By using an oil which is capable of separating from the plasticizer solution after it returns to ambient temperatures the oil will remain on the surface of the filament and not be absorbed into the filaments along with the plasticizer.

Generally the oil application is preferably applied to the cellulose acetate filaments by spreading the filaments out into a bend and spraying the spread-out filaments with a fine oily mist using a conventional paint spray gun. While this can be done either before or after the filaments are sprayed with plasticizer and dusted with powdered additives, the preferred procedure is as follows: After the filaments have been sprayed with a plasticizer solution containing the oil it is then dusted with the powdered additive. By this procedure there is a saving of processing steps. Accordingly, it is preferred to employ an oily material which is soluble in the plasticizer when heated but insoluble in the plasticizer when it is cooled back to

room temperatures. By using such a combination it is possible to apply the oil and plasticizer in one step. Such filters have the powdered additive strongly bonded to the filaments and, therefore, resist any sifting out of additive when jarred or tapped or otherwise subjected to movement which might tend to disturb the additive.

The amount of oil employed in the present invention to eliminate the sifting out of the powdered additive is preferably within the range of 3—8% based on the finished filter. This range is of some importance since less than 3% oil may only partly prevent the sifting out of the powdered additive and over 8% of the oil in the filter may cause its seepage through the paper wrapper used to wrap the filter in the manufacture of cigarette filters.

There are a number of oils which are satisfactory for bonding powdered additives to filament filters relatively strongly. As illustrative of these oils which also meet the other requirements of non-toxicity, freedom from odour, taste, and the like, are the following:—

A. Naturally occurring high-boiling liquid esters.

Examples: castor oil, cottonseed oil, almond oil, cocoanut oil, peanut oil, olive oil.

B. Liquid esters derived from natural fats.

Examples: glycerol oleates, glycerol ricinoleates, glycerol laurates.

C. Liquid high-molecular-weight fatty alcohols.

Examples: lauryl alcohol.

D. Mineral Oils.

Examples: Pharmaceutical grades of light, medium and high-viscosity mineral oils.

This form of the invention is illustrated in the following specific examples.

EXAMPLE 12.

Calcium Carbonate.

This example illustrates adding the oil as a separate step. Filter rods were produced as follows:—

Tow: 5 denier filament 70,000 total denier regular cross-section.

Plasticizer: Dimethoxyethylphthalate was sprayed on the tow at the approximate ratio of 20 parts plasticizer to 80 parts tow.

Powdered Additive: Calcium carbonate (average particle size, 5 microns) dusted on the plasticized tow at the ratio of 85 parts tow to 15 parts calcium carbonate.

Mineral Oil: 5% mineral oil (based on total weight) was sprayed on the plasticized tow containing the powdered additive.

This produced filter rods of 90 mm. in length with an average pressure drop equal

to 9.5 inches of water and a circumference of 25.2 mm. No calcium carbonate powder "sifted out" of the rods when they were held vertically and tapped vigorously against a hard, flat surface. Smoking experiments were carried out using standard American cigarettes containing 15 mm. filters cut from these rods. The tests indicated that 37% of the tars and nicotine were removed from the main stream smoke by the filters. The pressure drop of the filtered cigarettes was only 4% higher than that of the unfiltered cigarettes of equal length.

EXAMPLE 13.

Calcium Stearate. $\text{Ca}(\text{C}_{18}\text{H}_{35}\text{O}_2)_2$

This example illustrates the addition of a hot plasticizer soluble oil to the filter to prevent the "sifting out" of a powdered additive.

Five denier filament 70,000 total denier regular cross-section tow was sprayed with a hot (60° C.) solution of 25 parts glycerol monoleate and 75 parts triethyl citrate at the approximate ratio of 76 parts of tow to 24 parts of hot plasticizer-oil mixture. The treated tow was then dusted with calcium stearate powder (capable of passing through a No. 300 screen, U.S. sieve series) at the ratio of 80 parts treated tow to 20 parts powder. The powder did not sift out of the finished rods when they were tapped in the manner previously described. The rods (90 mm. in length) had an average pressure drop of 9.6 inches in water. Smoking experiments indicated that 15 mm. filters of these rods attached to the standard cigarettes removed 39% of the nicotine and tars from the main stream smoke. The filtered cigarette had a pressure drop which was only 6% higher than that of the unfiltered cigarette (standard cigarette) of equal length.

EXAMPLE 14.

Calcium Carbonate.

A 5 ft. length from a crimped continuous tow of cellulose acetate yarn containing 14,000 filaments of 5 denier/filament was fluffed out to a width of 12 inches. While in this fluffed out form it was sprayed on both sides with a hot solution consisting of 60 parts glycerol triacetate and 40 parts castor oil by weight. During the spraying operation the solution in the spray gun was maintained at 75–80° C. to keep the castor oil dissolved in the plasticizer. It was observed that immediately after the solution was ejected from the gun it formed a fine mist of plasticizer and castor oil particles. After being sprayed the spread out tow was dusted on both sides with calcium carbonate powder which had been sifted through a No. 325 screen, U.S. Sieve Series.

Analyses of the sprayed and dusted tow at this point showed that it consisted of 63% cellulose acetate yarn, 12% glycerol triacetate, 8% castor oil and 17% calcium carbonate powder by weight. The tow was pulled back into its original form of a cord or bundle and passed through a device which paper wrapped it and cut it into filter rods 90 mm. in length and 25.5 mm. in circumference. After a storage time of 24 hours at room temperature the rods were quite rigid due to the fusion action of the plasticizer on the cellulose acetate filaments. Some of the rods were opened and examined under the microscope. This revealed that the calcium carbonate particles were uniformly distributed on the surface of the filaments in the tow. It also revealed that the plasticizer had been absorbed by the filaments forming a fusion of the filaments at random spots along the entire length of the rod while the castor oil had spread over the surface of the filaments forming a tacky coating which prevented the calcium carbonate particles from sifting out of the rod.

Two of the rods were cut into 15 mm. filter tip lengths. These were attached to 10 standard king-size cigarettes which had been shortened by 15 mm. to compensate for the length of the filter. The filtered cigarettes were smoked to butt lengths of 35 mm. on the automatic smoking machine and the smoke which passed through them was collected and analyzed for nicotine and tar content.

Control filters (15 mm.) without the calcium carbonate additive were also prepared from another sample of the same tow used in preparing the filters with the additive. This sample had also been sprayed with the hot solution of 60 parts glycerol triacetate and 40 parts castor oil until the combination consisted of 75% cellulose acetate filaments, 15% glycerol triacetate and 10% castor oil. The control filters were attached to the same brand of standard king-size cigarettes, shortened by 15 mm. These were also smoked to butt lengths of 35 mm. on the automatic smoking machine and the smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content.

Ten unfiltered king-size cigarettes (same brand) which were not shortened by 15 mm. were smoked to butt lengths of 35 mm. on the automatic smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content. The results obtained from the three sets of cigarettes are listed below.

		<i>Av. Pressure Drop*</i>	<i>Mg. Tars in the Smoke</i>	<i>Mg. Nicotine in the Smoke</i>
	Unfiltered cigarette	3.3"	167	25
5	Cigarettes with cellulose acetate filter containing glycerol triacetate and castor oil	3.9"	140	21
	Cigarettes with cellulose acetate filter containing calcium carbonate, glycerol triacetate and castor oil	4.1"	100	16

10 * Pressure drop expressed as inches of water at an air flow rate of 17.5 cc./second through the cigarette.

From the results listed above it can be seen that the filter containing the calcium carbonate reduced the amount of tar collected by 40% and the amount of nicotine collected by 30%. The filter without the calcium carbonate reduced the amount of tar collected by 16% and the amount of nicotine collected by 16%. The increase in pressure drop of the filtered cigarette due to the addition of the calcium carbonate to the filter was only 4.9%.

It is believed apparent from the foregoing description it may be seen that the present invention also provides a relatively simple and economical way of improving the bonding of powdered additives to filament filters. By combining the oil and the plasticizer in one spraying operation, this operation can be carried out in the plasticizer booth without the necessity of installing added equipment.

In carrying out the operation of this invention, the purity of the carbonate or phosphate employed is not a critical factor. Rather, the form of its particles is more important. The salt should be in a finely powdered form so that it can be readily slurried in a liquid or passed through a spraying nozzle. While the exact size of the particle does not appear to be critical, it is indicated that the salt additive should be at least capable of passing through a No. 200 screen (U.S. Sieve Series). Commercial grades of the salts of the invention generally may be obtained in a particle size which will pass through a No. 325 screen (U.S. Sieve Series). Advantageously, a salt is used which has at least 80% of its particles of a diameter less than the diameter of the filaments of the filter. For filaments of the size used in the example above, no substantial number of particles should be as large as twice the filament diameter or as small as one-tenth the filament diameter although these limits may vary considerably with filaments of other sizes. Any source of salt which passes this requirement can be employed.

While unusually good results have been obtained with filters prepared from a crimped continuous filament tow of cellulose acetate or regenerated cellulose filaments, it will be recognised by those skilled

in the art that the usefulness of the invention extends to other fibrous filters, including those prepared from continuous filaments of nylon, polyamides, polyesters, etc., particularly where the filaments are in substantial longitudinal alignment and are co-extensive with the body of the filter. It is preferred to use for filter preparation in accordance with the present invention a crimped continuous strand, e.g. tow, of 4,000 to 35,000 filaments, the filaments being of 16 to 3 denier and having about 4 to 10 crimps per inch.

By the expression "surface solvation" as used herein is meant the creation, by the action of a solvent or plasticizer and/or heat, of an adhesive, tacky or readily bonding condition of the filaments by solution or incipient solution of surface portions of the filament material whereby there is produced a welding and adhesion between adjacent filaments contacting at such portions, and by "coalescence" is meant the situation caused by partial or incipient solvation of surface portions of the filaments and resulting in a condition within those portions under which the portions will flow into or unite with similar portions of dissolved or plastic material in contiguous filaments. Surface solvation bonds between fibres are sometimes also referred to as "fused" or "welded" bonds.

It is to be noted that while the filters of the present invention derive special benefits from the chemical properties and behaviour of the water-insoluble inorganic salt additives, further advantages can be obtained from the following structural factors found in the most advantageous form of the invention:—

1. That the aligned filaments be randomly bonded at randomly spaced intervals. This may be achieved by spraying a solvent type of plasticizer on to a spread tow of synthetic thermoplastic filaments as described above. It also may be accomplished by use of a filament strand in which at least some of the filaments contain sufficient plasticizer to be softened at slightly elevated temperatures. This plasticizer may be added by spraying it on to the formed filaments or it may be added to the spinning solution before the filaments are spun.

2. That where the additive is to be applied before or with a filament bonding agent, no material should be employed for bonding the filaments which would coat, destroy or otherwise substantially interfere with the activity or integrity of the salt particles. That is to say, in such a case the filament bonds should be formed by coalescence of the surface solvation type. Obviously no plasticizer should be employed which has a detrimental action on the salt particles.

3. That substantially all the salt particles be carried on the surfaces of the filaments, supported by the filaments, and substantially immobile with respect thereto. This structure is best accomplished by softening of portions of the filament surfaces with a solvent or by creating a superficial surface with a film-forming material whereby the particles may be slightly embedded in the surface or superficial surface without being coated or covered. It is also most easily accomplished when the diameters of the particles are not too different from the diameters of the filaments, the major portion of the particles being, for instance, less than twice the filament diameter, but more than one-tenth of the filament diameter.

30 WHAT WE CLAIM IS:—

1. A tobacco smoke filtering element comprising a bundle of continuous synthetic filaments having finely divided particles of a water-insoluble calcium or magnesium salt distributed over the surfaces of the filaments and bonded thereto by means of a bonding agent compatible with the material of the filaments.

2. A tobacco smoke filtering element according to Claim 1, wherein the salt is the salt of an acid which is weaker than acetic acid.

3. A tobacco smoke filtering element according to Claim 2, wherein the salt comprises a phosphate or carbonate of calcium or magnesium.

4. A tobacco smoke filtering element according to Claim 2, wherein the salt comprises CaCO_3 , MgCO_3 , $\text{Mg}_3(\text{PO}_4)_2$, $\text{Ca}_3(\text{PO}_4)_2$, $\text{Ca}_2\text{P}_2\text{O}_7$, CaHPO_4 or $\text{Ca}(\text{C}_2\text{H}_3\text{O}_5)_2$.

5. A tobacco smoke filtering element according to any of the preceding claims, wherein the finely divided particles of the salt are smaller than No. 200 (U.S. Sieve Series).

6. A tobacco smoke filtering element according to any of the preceding claims, wherein the bonding agent and particles of salt both comprise more than 10% by weight of the element.

7. A tobacco smoke filtering element according to any of the preceding claims, wherein the filaments comprise crimped,

continuous cellulose acetate filaments which are bonded to one another at random points of contact by means of a plasticizer which also serves as the bonding agent for the particles.

8. A tobacco smoke filtering element according to Claim 7, wherein the cellulose acetate has an acetyl content of 39.2%.

9. A tobacco smoke filtering element according to Claim 7 or 8, wherein the bundle of continuous crimped cellulose acetate filaments comprises a tow of 4,000 to 35,000 filaments, the filaments being of 16 to 3 denier per filament and having 4 to 10 crimps per inch.

10. A tobacco smoke filtering element according to Claim 7, 8 or 9, wherein the plasticizer comprises a phthalate, citrate or propionate.

11. A tobacco smoke filtering element according to Claim 9 or 10, wherein at least 80% of the finely divided particles of the salt have a diameter less than the diameter of the continuous synthetic filaments and no substantial number of the particles have a diameter as large as twice the filament diameter or as small as one-tenth the filament diameter.

12. A tobacco smoke filtering element according to any of the preceding claims wherein the filaments carry on the surfaces thereof an oily material.

13. A method of making tobacco smoke filtering elements comprising the steps of opening a tow of continuous synthetic filaments, spraying the spread filaments with a bonding agent compatible with the material of the filaments, distributing over the surfaces of the filaments finely divided particles of a water-insoluble calcium or magnesium salt, such particles being bonded to the surfaces of the filaments by the bonding agent, and compacting the filaments into a filter rod.

14. Method according to Claim 13, wherein an oily material is applied to the spread filaments prior to the distribution of the particles over the filaments.

15. Method according to Claims 13 and 14, wherein the bonding agent comprises a plasticizer for the material of the filaments and is sprayed on to the spread filaments in admixture with the oily material.

16. Method according to Claim 15, wherein the oily material is insoluble in the plasticizer at ambient temperatures but soluble therein at elevated temperatures, the mixture being sprayed on to the spread filaments at an elevated temperature so that the oil remains on the surfaces of the filaments and the plasticizer is absorbed therein.

17. Method according to any of the preceding Claims 13 to 16, wherein the finely divided particles of the salt are distributed

over the spread filaments as a slurry in the filtering elements substantially as herein-
bonding agent. before described.

18. Tobacco smoke filtering elements
substantially as hereinbefore described.

L. E. T. BRANCH, B.SC., F.R.I.C.,
Patent Agent.

5 19. Methods of making tobacco smoke

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Tobacco smoke filter

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Publication date: 1967-06-28

Inventor(s):

Applicant(s): PHILIP MORRIS INC +

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more >>

Abstract of **GB 1073896 (A)**

A tobacco smoke filter comprises synthetic filaments, activated carbon particles and a high molecular weight (i.e. between 600 and 20,000) polyalkalene glycol. Preferably the polyalkalene glycol is a polyethylene glycol, a methoxy polyethylene glycol, or a propylene glycol. The activated carbon particles have a mesh size (U.S. Standard) of 10-325, preferably 10-45, and the filter may contain from 2 to 200 parts by weight of carbon particles and from 2 to 25 parts by weight of glycol per 100 parts by weight of filaments. Suitable synthetic fibres are cellulose acetate tow or yarn having a total denier of 25,000-75,000 and a denier per filament of 1-16. Flavourants may be included in the filter. The filters, which may be used in series with conventional filters, may be prepared by spreading the fibres and coating them, e.g. by spraying, with the polyalkalene glycol, after which the carbon particles are applied to the coated fibres. Before the glycol has solidified the fibres are formed into a rod. Quantitative examples are given.

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PATENT SPECIFICATION

DRAWINGS ATTACHED

1073,896

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Date of Application and filing Complete Specification: May 7, 1965.

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ERRATUM

SPECIFICATION No. 1,073,896

Page 7, line 88, for "grams sample X 200"
read "grams sample X 2000"

THE PATENT OFFICE
25th March 1968

esters, have found widespread use in tobacco smoke filters due to the ease with which they can be manufactured into filter rods on standard cigarette manufacturing equipment. 20 These synthetic fibers generally comprise cellulose acetate in the form of crimped, continuous fibers or filaments. Filters made of cellulose ester fibers function, in general, by removing a portion of the particulate matter 25 from the smoke which passes through the fibers. The crimping or other physical positioning of the fibers within the filter serves to increase the surface area of the filaments which come in contact with the smoke. However, filters consisting of such fibers alone do 30 not remove any significant amounts of undesirable gas phase constituents from the tobacco smoke.

Certain materials have also been applied 35 to synthetic fibers which are used in filters in order to make the fibers more effective in picking up the particulate matter in the smoke. Such materials may, for example, be starch, ground cellulose or the like and are 40 generally held in place by an adhesive or plasticizer such as dimethoxyethylphthalate, methylphthalylethylglycollate or triacetin. However, these materials do not materially alter the failure of the fibers to remove un-

by activated carbon, have also been employed in tobacco smoke filters. Such adsorbent particles have been employed in conjunction with crimped paper and serve to increase the effectiveness of such filters for removing the particulate matter in the smoke, as well as functioning to remove undesirable gas phase constituents from the smoke. However, when activated carbon has been used in this manner, it has been employed in the form of very fine particles or dust, because it had been found that larger carbon particles are not completely 70 satisfactory when incorporated with paper. The larger particles have been found to be virtually impossible to employ in paper-type filters.

Since paper-type filters have been found to be less efficient than synthetic fiber or tow-type filters, activated carbon particles have also been suggested for incorporation in synthetic filaments to form tobacco smoke filters. However, it has been generally recognized that the particles of carbon will not by themselves adhere well to the synthetic fibers in the filter and adhesives such as triacetin, dimethoxyethylphthalate or methylphthalylethylglycollate have been employed to hold the 85 particles on the fibers. Such adhesives have been found to poison or inactivate the activated carbon to a considerable extent.

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Index at acceptance: —A2 C(1E2, 1E3)

Int. Cl.: —A 24 c 5/50

COMPLETE SPECIFICATION

Tobacco Smoke Filter

- We, PHILIP MORRIS INCORPORATED, a corporation organised under the laws of the State of Virginia, United States of America, of 100 Park Avenue, New York 17, State of New York, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—
- This invention relates to tobacco smoke filters comprising synthetic fibers, activated carbon and a high molecular weight polyalkylene glycol.
- Synthetic fibers, for example, cellulose esters, have found widespread use in tobacco smoke filters due to the ease with which they can be manufactured into filter rods on standard cigarette manufacturing equipment.
- These synthetic fibers generally comprise cellulose acetate in the form of crimped, continuous fibers or filaments. Filters made of cellulose ester fibers function, in general, by removing a portion of the particulate matter from the smoke which passes through the fibers. The crimping or other physical positioning of the fibers within the filter serves to increase the surface area of the filaments which come in contact with the smoke. However, filters consisting of such fibers alone do not remove any significant amounts of undesirable gas phase constituents from the tobacco smoke.
- Certain materials have also been applied to synthetic fibers which are used in filters in order to make the fibers more effective in picking up the particulate matter in the smoke. Such materials may, for example, be starch, ground cellulose or the like and are generally held in place by an adhesive or plasticizer such as dimethoxyethylphthalate, methylphthalylethylglycollate or triacetin. However, these materials do not materially alter the failure of the fibers to remove undesirable gas phase constituents from the smoke.
- Other materials which are, in effect, self-bonding materials have been suggested for incorporation in synthetic fibers to be used in tobacco smoke filters. These materials include, for example, certain types of wax particles, such as paraffin or polyethylene wax, and certain sugar esters, such as sucrose acetate propionate. However, filters containing such additives also fail to remove any substantial amount of the undesirable elements which are present in the gas phase of the tobacco smoke.
- Certain adsorbent particles, as exemplified by activated carbon, have also been employed in tobacco smoke filters. Such adsorbent particles have been employed in conjunction with crimped paper and serve to increase the effectiveness of such filters for removing the particulate matter in the smoke, as well as functioning to remove undesirable gas phase constituents from the smoke. However, when activated carbon has been used in this manner, it has been employed in the form of very fine particles or dust, because it had been found that larger carbon particles are not completely satisfactory when incorporated with paper. The larger particles have been found to be virtually impossible to employ in paper-type filters.
- Since paper-type filters have been found to be less efficient than synthetic fiber or tow-type filters, activated carbon particles have also been suggested for incorporation in synthetic filaments to form tobacco smoke filters. However, it has been generally recognized that the particles of carbon will not by themselves adhere well to the synthetic fibers in the filter and adhesives such as triacetin, dimethoxyethylphthalate or methylphthalylethylglycollate have been employed to hold the particles on the fibers. Such adhesives have been found to poison or inactivate the activated carbon to a considerable extent.

Some of the disadvantages of incorporating carbon particles in synthetic filaments have been overcome by the use of an improved adhesive mixture comprising polyvinyl pyrrolidone and a polyhydric alcohol, such as glycerol. Such an adhesive has made it possible to employ carbon particles over a wider range of sizes than had previously been possible. However, even such an improved adhesive has had only limited effectiveness and it has been taught that for such applications the carbon particles should preferably be of such a size that no more than a very small percentage of them (most preferably no more than 5%) are larger than 48 mesh.

While it has also been proposed to employ larger particles of carbon in a filter, such particles have not been used in the fiber-containing section. Instead, such particles have been used in a separate filter section comprised solely of the carbon particles and this section has been placed between two non-carbon containing filter sections. This type of filter has an advantage over the previous filters in that the carbon particles need not be contacted with an adhesive to hold them on paper or tow. However, such filters create a different problem, in that the loose carbon particles tend to migrate from the section in which they are placed to other parts of the cigarette. Furthermore, this type of filter has the disadvantage that the carbon particles do not contact all of the tobacco smoke to the same degree. In such a filter, there is some settling of the carbon particles within the carbon-containing section of the filter during storage and transit of the cigarettes, creating an open space above the carbon. There is also a considerable amount of empty space around each of the carbon particles. Thus, a substantial percentage of the tobacco smoke, following a path of least resistance, can pass through these empty spaces in the filter without any significant contact between the smoke and the carbon. As will be more fully set forth below, the present invention overcomes this disadvantage and provides for intimate contact between substantially all of the tobacco smoke and the carbon particles.

Filters have also been made with adhesives to adhere the carbon particles together to form a disk or plug composed of carbon and adhesive. While such construction tends to overcome the problem of carbon particle migration, the adhesives in such filters have been found to have been employed in such a manner that the carbon is at least partially deactivated by the adhesives. Furthermore, such filters have also been found to give relatively poor contact between the smoke and the carbon particles.

In one aspect the invention provides a filter for tobacco smoke comprising synthetic filaments or fibres, activated carbon particles, particularly activated carbon particles of rela-

tively large size, and a high molecular weight polyalkylene glycol (as hereinafter defined).

The high molecular weight polyalkylene glycol, which will be described in more detail hereinafter, serves to selectively remove certain gaseous phase components from the tobacco smoke as well as to bind the carbon particles to the filtering medium without adversely affecting the character or the quality of the smoke which passes through the filter. In addition, the high molecular weight polyalkylene glycols are compatible with the materials generally employed in filter manufacture and do not have any objectionable taste or odour.

The invention is illustrated in the accompanying drawings wherein.

Fig. 1 is a longitudinal sectional view of a cigarette embodying the filter of the present invention.

Fig. 2 is a cross-sectional view of the cigarette shown in Fig. 1, taken along the lines 2—2.

Fig. 3 is a longitudinal sectional view of a modification of the present invention.

Fig. 4 is a cross-sectional view of the cigarette shown in Fig. 3, taken along the lines 4—4.

Fig. 5 is a magnified view of a portion of the cigarette shown in Figs. 1 and 2, showing in greater detail the body of filtering material prepared in accordance with the present invention.

Referring to Figs. 1 and 2, there is shown a cigarette 1 having a paper cylinder 2. Shredded tobacco 3 is contained within paper cylinder 2. Paper cylinder 4 is positioned at the other end of the cigarette from the shredded tobacco and abuts one end of paper cylinder 2. Paper cylinder 4 generally defines the filter unit of cigarette 1. Paper cylinder 4 contains a carbon-free filter section 5, which is preferably tow, which is contained within paper cylinder 6. Carbon-containing section 7 contained within paper cylinder 8 comprises cellulose acetate filaments 9 and carbon particles 10 which are held in place by high molecular weight polyethylene glycol adhesive 11. The filter unit, which is defined by paper cylinder 4, is joined to the filler section, which is defined by paper cylinder 2, by paper cylinder 15, which covers all of paper cylinder 4 and a portion of paper cylinder 2.

Figs. 3 and 4 illustrate another form of the present invention wherein there is shown a cigarette 10 having a paper cylinder 20. Shredded tobacco 30 is contained within paper cylinder 20. Paper cylinder 40 is positioned at the other end of the cigarette from the shredded tobacco and abuts one end of paper cylinder 20. Paper cylinder 40 generally defines the filter unit of cigarette 10. Paper cylinder 40 contains a carbon-free filter section 50, which is preferably tow, which is contained within paper cylinder 60. Carbon-

containing section 70 contained within paper cylinder 80 comprises cellulose acetate filaments 90 and carbon particles 100 which are held in place by high molecular weight polyethylene glycol adhesive 110. Carbon-free section 55 contained within paper cylinder 56 is positioned between section 70 and filler 30 and serves to isolate the carbon-containing section from the filler as well as to provide additional particulate matter filtration. The filter unit, which is defined by paper cylinder 40, is joined to the filler section, which is defined by paper cylinder 20, by paper cylinder 150, which covers all of paper cylinder 40 and a portion of paper cylinder 20.

With particular reference to Fig. 5, which illustrates in detail a preferred form of the present invention, cellulose acetate fibers 9 are shown as being substantially continuous, crimped filter elements which are substantially longitudinally aligned and spaced to provide passage for tobacco smoke therethrough. Relatively large carbon particles 10 are positioned within the spaces between fibers 9 and are held in position by high molecular weight polyethylene glycol adhesive 11.

It will be seen from Fig. 5 that each particle of carbon generally extends within several of the channel-like spaces which are formed by the cellulose acetate filaments. These channel-like spaces generally define a plurality of tortuous paths along which the tobacco smoke travels. During such travel, the tobacco smoke encounters the relatively large particles and is diverted thereby through adjacent passages. In this manner, the smoke not only passes around each particle with substantial contact occurring but also is forced through a more complex passage through the filaments themselves. Thus, the smoke is forced into more intimate contact with both the carbon particles and cellulose acetate fibers than has heretofore been possible.

It will also be noted from Fig. 5 that some of the particles of high molecular weight polyethylene glycol adhere to the cellulose acetate fibers as distinct particles and that such particles do not hold any carbon in place by direct contact with the carbon. These particles also serve to enhance the filtering ability of such a filter, since they provide additional barriers to the passage through the filter of the particulate matter in the smoke. In addition, the high molecular weight polyethylene glycol itself serves to remove phenols and other undesirable gas-phase constituents from the tobacco smoke.

It will be understood that the materials employed in the cigarettes shown in the drawing is merely illustrative. For example, the form of carbon particles shown and the specific polyalkylene glycols described are merely representative of the carbon particles and polyalkylene glycols which may in general be used. Likewise the paper coverings for the

respective filter sections may also be varied from the arrangements shown in the drawing, dependent upon various factors, such as manufacturing convenience.

In order to prepare the tobacco smoke filter of the present invention, the cellulose acetate fibers or similar fibers are preferably spread out and thereafter sprayed with a mist containing the high molecular weight polyalkylene glycol. In this manner there is formed on the fibers a non-continuous covering comprising small particles of the high molecular weight polyalkylene glycol.

The high molecular weight polyalkylene glycol may be sprayed or applied to the fibers by means of a centrifugal applicator. In order to accomplish this, the high molecular weight polyalkylene glycol must be in a flowable form. This can be accomplished by heating the high molecular weight polyalkylene glycol to a temperature slightly higher than its melting point. It is then fed to a rotating perforated cylinder, from which it is applied to the fibers in the form of a fine mist. Alternatively, it may be sprayed by spray equipment. It may also be dissolved in water and applied as a solution, if desired, or by other suitable means.

While the above method for applying the high molecular weight polyalkylene glycol is preferred, it may also be deposited on the fibers as a film, for example, a continuous or semi-continuous coating.

For convenience, the fibers after the high molecular weight polyalkylene glycol has been applied will be referred to as "coated fibers", regardless of whether the glycol was applied by centrifugal applicator, by spraying or otherwise, and regardless of whether the glycol is applied as discrete particles, as a non-continuous coating or as a continuous coating.

After the application of the high molecular weight polyalkylene glycol, the carbon particles are applied to the coated fibers.

Some of the high molecular weight polyalkylene glycols which may be employed will not completely harden after being applied to the fibers and will remain on the fibers, for example, as extremely viscous liquids. In this case their adhesive action in that state is such that the carbon can be added either immediately after the coating is applied or at a later time, even after standing at room temperature.

High molecular weight polyalkylene glycols, which must be heated to make them flowable, will, upon cooling, harden after application to the fibers. In this case, the activated carbon particles should be dispersed over the coated fibers before the high molecular weight polyalkylene glycol particles have solidified on the fibers. Obviously, the coated fibers can, where necessary, be maintained or placed in an atmosphere which is at a sufficiently high temperature so that the high molecular weight polyalkylene glycols possess tacky or adhesive

qualities when the carbon particles are applied.

The carbon-containing coated fibers, still before the polyalkylene glycol has solidified, may then be gathered together into the desired configuration for a filter and the resulting material formed within a paper cylinder or other wrapper to be held in the desired shape. The forming operations may be those conventionally employed in the manufacture of cigarette filters.

The synthetic fibers which may be employed in accordance with the present invention are preferably cellulose acetate fibers and may be cellulose acetate tow or yarn having a total denier of from about 25,000 to about 75,000, and preferably from about 30,000 to about 50,000, and having a denier per filament of from about 1 to about 16, and preferably from about 3 to about 6. Preferably, the cellulose acetate fibers or filaments are generally longitudinally aligned and coextensive with the length of the filter element and have portions thereof which are crimped into a different alignment from the general orientation of the filaments, whereby the filaments have certain portions which somewhat intermesh, as illustrated in the drawing. While cellulose acetate fibers are the preferred materials, other cellulose esters, for example, cellulose butyrate, or other types of synthetic fibers and mixtures of synthetic fibers may also be employed as the fibers of the present invention.

The activated carbon particles which are

employed in accordance with the present invention may have a particle size of from about 10 to about 325 mesh (United States Standard) but preferably are relatively large particles of from 10 to 45 mesh size, i.e., substantially all of the particles will pass through a 10 mesh screen and will be retained on a 45 mesh screen. Most preferably, at least 95% of the particles should be retained on a 30 mesh screen and should pass through a 12 mesh screen. The particles may be roughly spherical in shape or may have other regular or irregular shapes, depending on how they are produced. Preferably, they have irregular shapes, such as are produced by grinding coconut-type carbon.

The activated carbon which is employed may be one of various known types, such as coke base carbon or nutshell carbon, or any other suitable carbon.

Preferably, the carbon employed is a coconut-type carbon. Such carbons have a somewhat irregular shape, and are therefore held somewhat more easily in position within the mass of synthetic filaments. They also afford good contact and good pick up of undesirable gas phase constituents.

A particularly preferred carbon is a coconut carbon such as the PCB grade activated charcoal manufactured by Pittsburgh Coke and Chemical Company. This carbon has a high rate of adsorption and a high retentivity. As one specific example, the carbon may have the following properties:

70	Physical Properties	
	Total Surface Area Sq. Meters/Gram	1200
	Apparent Density	27.5 lbs./Ft. ³
	Pore Volume (within Particle)	+9.4 CC/Gm.
	Voids in Dense Packed Column %	38—40
75	Specifications	
	Iodine No. Mg./Gram	1200 Min.
	Carbon Tetrachloride Adsorption Wt. %	60% Min.
	Ash—Maximum %	4%
	Moisture—Max. % as Packed	3%
80	Hardness Number, Min.	92 Min.

Size Breakdown—12×30 Mesh

	Sieve No.	% Retained
85	+12	0—5
	12×16	20—40
	16×20	40—85
	20×30	10—20
	—30	0—5

The high molecular weight polyalkylene glycol may be polyethylene glycols, polypropylene glycols, glycols containing both $-C_2H_4O-$ and $-C_3H_6O-$ groups and may also be mono- or di-esters of such high molecular weight polyalkylene glycols, for example, the methoxy esters of polyethylene

glycols. As used herein, the term "high molecular weight polyalkylene glycol" is intended to include all such materials, including the esters, having molecular weights of from about 600 to about 20,000.

Illustrative of such materials are polyethylene glycols having the general formula:



wherein x is an integer having a value of from about 12 to about 453. Such materials may be prepared by conventional methods. For example, 2 moles of the monosodium salt of diethylene glycol of beta, beta'-dichloroethyl ether can be condensed to yield hexaethylene

- glycol dichloride, which is then condensed with 2 moles of the monopotassium salt of hexaethylene glycol to yield an 18 membered compound. The synthesis may be continued in this manner to yield even high molecular weight compounds.
- Examples of suitable polyalkylene glycols include materials sold under the trade name Carbowax. The carbowax polyethylene glycols are commonly referred to in abbreviated form. For example, Carbowax 600 is the abbreviated form for Carbowax brand polyethylene glycol 600.
- Carbowax 600 is a preferred adhesive material. This material has an average molecular weight of about 600 and is a soft, white, waxy substance at room temperature.
- Illustrative of other Carbowax materials which may be employed are: Carbowax 1000, Carbowax 1500 W, Carbowax 1540, Carbowax 4000 and Carbowax 6000. These materials have the following properties:
- Carbowax 1000. Average molecular weight 950—1050. Soft, white, wax solid. Solidifying range 35—40°C. Flash pt. >450°F. Saybolt viscosity at 210°F., sec. 70 to 100. Solubility in water at 20° approx. 70% w/w. Comparative hygroscopicity=5 (glycerol=100).
- Carbowax 1500 W. Average molecular weight 500 to 600. Soft, white, waxy solid having the consistency of low-melting petrolatum. Specific gravity at 20°C (relative to water at 20°C) 1.151. Solidifying range 35 to 40°C. Flash pt. 430°F. Solubility in water at 20° approx. 73% w/w. Completely soluble in water at 50°C. Insoluble in petr. ether. The pH of a 5% aq. soln. is about 4.6. Comparative hygroscopicity=30 (glycerol=100). Saybolt viscosity at 210°F., sec. 60 to 90. LD₅₀ i.p. in mice: 9.2 g./kg.
- Carbowax 1540. Average molecular weight 1300—1600. Soft, white, waxy solid. Specific gravity at 20°C (relative to water at 20°C) 1.15. Solidifying range 40—50°C. Flash pt. >450°F. Solubility in water at 20°C. approx. 70% w/w. Insoluble in petroleum ether. The pH of a 5% aq. soln. is about 6.5. Comparative hygroscopicity=5 (glycerol=100). Saybolt viscosity at 210°F., sec. 100 to 150.
- Carbowax 4000. Average molecular weight 3000 to 3700. Hard, white, waxy solid. Specific gravity at 20°C (relative to water at 20°C) 1.204. Solidifying range 50—55°C. Flash pt. 475°F. Solubility in water at 20°C. approx. 62%. Insoluble in petroleum ether. The pH of a 5% aq. soln. is about 6.35. Comparative hygroscopicity=1 (glycerol=100). Saybolt viscosity at 210°F., sec. 400 to 800.
- Carbowax 6000. Average molecular weight 6000—7500. Hard, white, waxy solid. Solidifying range 58—62°C. Flash pt. >475°F. Solubility in water at 20°C. approx. 50% w/w. Insoluble in petr. ether. Comparative hygroscopicity=1 (glycerol=100). Saybolt viscosity at 210°F., sec. 6000—7500.
- Alkoxy polyethylene glycols may also be employed, for example, methoxy polyethylene glycol or other lower alkoxy polyethylene glycols. A specific illustration of such material, and a preferred material in this invention, is Carbowax methoxy polyethylene glycol 750 which has the following properties: Average molecular weight of 715—785; specific gravity at 40/20°C. of 1.094; viscosity CKS at 210°F. of 10.5 and freezing range of 27 to 32°C. This material has been found to give even better results than Carbowax 600.
- High molecular weight polypropylene glycols and mixed glycols having similar properties to the high molecular weight polyethylene glycols may also be employed.
- The Carbowaxes are more completely described in the publication of Union Carbide Chemicals Company entitled "Carbowax Polyethylene Glycols".
- In filter plugs which embody the present invention, from about 2 to 200 parts, by weight, of carbon and from about 2 to 25 parts by weight of high molecular weight polyalkylene glycol are employed per 100 parts of fiber. Preferably, when the filter plug is to be employed in a cigarette or similar product, sufficient carbon should be present to provide from about 2 to about 150 mg. of carbon per cigarette. Most preferably, the amount of carbon is about equal, for example, no more than 20%, by weight, higher or lower, to the combined weight of tow, paper and paste employed in the filter plug. The amount of high molecular weight polyalkylene glycol employed, under such circumstances, will preferably be about 0.1% to about 0.3%, by weight, of the amount of carbon employed.
- Filter plugs embodying the present invention may contain other additives, for example other adsorbents, such as silica gel, rice starch, activated aluminas and the like, but preferably contains the high molecular weight polyalkylene glycol as the sole adhesive or binding agent.
- Filter plugs embodying the present invention will generally be from about 4 to 25 mm. in length, when employed in cigarettes or the like. The plugs are preferably incorporated in combination with an outer carbon-free plug, which may be a paper type plug or a tow type plug. Preferably, the outer plug is a cellulose acetate crimped fiber type plug having a total denier of from about 25,000 to 100,000, a denier per fiber of from about 1 to 16, a plug length of from about 4 to 20 mm. and a maximum RTD (resistance to draw) of about 6 inches of water.
- An inner plug positioned between the tobacco and the carbon-containing plug may also be employed, if desired. This inner plug

may be similar in nature to the outer plug.

Both the inner plug and the outer plug, when employed, may have incorporated therein high molecular weight polyethylene glycols, triacetin, various waxes, and other additives and similar materials and may also include various flavorants, if desired.

It is to be understood, however, that when an inner plug and an outer plug are employed in combination with a carbon-containing filter of the present invention, the inner and outer plugs need not be of the same construction or contain the same additives, if any. Thus, for example, the outer plug may contain various flavorants which are not present in the inner plug.

For a further understanding of the present invention, reference may be had to the following examples, which merely serve to illustrate certain forms of the present invention.

EXAMPLE 1

A 5 denier per filament cellulose acetate yarn in the form of crimped tow containing 40,000 filaments was pulled over a series of air jets which spread the filaments over a width of about 8—12 inches. While in this spread-out condition, the filaments were passed through a centrifugal applicator wherein a high molecular weight polyethylene glycol (Carbowax 600) which has been maintained in a reservoir at a temperature of from 30 to 35°C. (i.e., about 5—10°C. above its melting point) is sprayed on both the upper and lower surfaces of the spread-out tow maintained at about the same temperature as the Carbowax 600. Other high molecular weight polyalkylene glycols are also preferably applied to tow, in accordance with the invention, at a temperature slightly above their melting point, for example, about 10°C. above their melting point.

The high molecular weight polyethylene glycol is applied to the fibers in the form of small droplets. The resulting coated spread-out tow is then passed beneath a hopper through which particles of PCB type carbon, having a particle size of 12 to 30 mesh are passed, whereby the carbon particles are uniformly distributed across the surface of the spread-out and coated fibers. The carbon particles are fed by means of a standard magnet vibratory feed unit. The resulting material is then fed through a plenum chamber and is thereafter passed through a device whereby the tow is formed into a cylindrical shape and is wrapped with a paper wrapper to produce a continuous rod with a circumference of about 25 mm.

The resulting rod is then ultimately cut into 12.5/ mm. filter tip lengths which gradually become firm due to the adhesive action of the high molecular weight polyethylene glycol on the cellulose acetate fibers and on the carbon particles. Analyses of this filter

section from which the paper wrapper was removed show that it consisted of about 48% cellulose acetate filaments, 3% high molecular weight polyethylene glycol and 49% activated carbon.

The activated carbon was not readily dislodged from the fibers even with vigorous tapping of the filter section against a hard surface.

Examination of a number of such filter sections indicated that the carbon particles extend in general into more than one of the paths formed by the cellulose acetate filaments and that the carbon particles were held in place by means of the Carbowax 600, particles of which were also deposited on the filaments apart from the carbon particles. The Carbowax 600 served to hold the carbon particles in place by two actions: (1) it bound the cellulose acetate filaments together to form a web which held the carbon particles firmly in place, the size and shape of the particular coconut carbon employed facilitating this holding action and (2) it directly bound carbon particles to the fibers by adhesive action.

Ten of these filter tips were each separately attached to ten outer filter plugs of 4.5 denier cellulose acetate tow containing 68,000 filaments. These outer plugs were 7.5 mm. long and contained 10% of a plasticizer comprising 50% Carbowax 600 and 50% triacetin. The two filter sections, in each case, were attached to a standard tobacco mixture contained in a length of cigarette paper sufficient to give an over-all cigarette length of 85 mm. The outer plug also contained selected flavorants.

The cigarettes were found to have the following properties (based on an average of the ten cigarettes):

- (1) Filter efficiency (% tar removed) 50.1%
- (2) Total RTD 4.56 inches of water
- (3) Inner plug RTD 2.57 inches of water
- (4) Outer plug RTD 1.09 inches of water

A commercially available filter cigarette of the cellulose acetate type and a commercially available cigarette having a filter section comprised of free particles of carbon were tested in comparison with the cigarettes prepared in accordance with this invention with the following results: the two commercially available cigarettes had lower efficiencies (41.4 and 46.4%, respectively) than the present cigarettes and, when tested by chromatographic determinations for the removal of gas phase components, the present filters were found to remove from the smoke greater amounts of most of the undesirable gas phase constituents, such as acetaldehyde, acrolein, methyl ethyl ketone, acetonitrile, benzene and the like.

Cigarettes were prepared in substantially the same manner, employing (separately) in place of Carbowax 600, Carbowax 750, Carbowax 1000 and Carbowax 1540, with similar results to those obtained using Carbowax 600. Be-

cause of their more solid consistency, these higher molecular weight materials are preferred over the Carbowax 600.

EXAMPLE 2

5 Cigarettes prepared in a similar manner to Example 1 were prepared in two batches: one batch had carbon plugs containing the same amount of Carbowax 600 as the cigarette of Example 1 and the second batch had
10 carbon plugs containing no Carbowax 600. Except for the Carbowax 600, the cigarettes were substantially identical. Smoke from these cigarettes was analyzed. It was found that the Carbowax-containing cigarettes removed about
15 18% more phenols than did the Carbowax-free cigarettes, with all other efficiencies and removals of the cigarettes tested being approximately the same for each type of cigarette.

20 Resistance to draw, also referred to in this specification as RTD, was determined as follows:

A vacuum system was set to pull an air flow of 1050 cc./min. by inserting the tapered end of a standard capillary tube through the dental dam of the cigarette holder and adjusting the reading on the water manometer to the correct RTD. The water level of the manometer was set at zero before inserting the standard capillary.

30 Then, the butt end of a cigarette or plug was inserted to a depth of 5 mm. in the dental dam of the cigarette holder. The pressure drop behind this cigarette with 1050 cc./min. of air flow passing through was read directly as RTD (inches water) from the inclined water manometer.

35 The molecular weight described in this specification is an average molecular weight and is determined as follows:

40 (1) Preparation of phthalic anhydride-pyridine reagent: Add 42 g. of phthalic anhydride to 300 ml. of freshly distilled pyridine

contained in a one-quart brown glass-stoppered bottle. Shake the bottle vigorously until complete solution is effected. The reagent preferably should stand overnight before using; however, the solution may be heated under hot tap water until a slight cooling of the reagent occurs, indicating complete reaction.

(2) Procedure: Prepare a sufficient number of clean dry heat-resistant pressure bottles to make all control and sample determinations in duplicate. Carefully pipet 25 ml. of the phthalic anhydride-pyridine reagent into each of the bottles, using the same pipet for each transfer. Reserve two of the bottles as blanks.

Into each of the other bottles introduce the specified amount of sample in the manner indicated. Make all weighings to the nearest 0.1 mg.

Fit the bottles with pressure stoppers and wrap each securely in a canvas bag. Place the samples and blanks as close together as possible in a water bath maintained at $98 \pm 2^\circ\text{C}$. for 30 minutes. Maintain sufficient water in the bath to just cover the liquid in the bottles. Remove the bottles from the bath and allow them to cool in air to room temperature. Do not remove the wrappers from the bottles while they are hot, or attempt to hasten the cooling by immersing them in cold water, as a serious accident can result from the breakage of the bottles. When the bottles have cooled, loosen the wrappers, uncup to release any pressure, and then remove the wrappers.

To each bottle add exactly 50 ml. of standard 0.5 N sodium hydroxide, using the same pipet for each addition. Be sure to allow the same drainage time for each bottle as this amount is not considered in the final calculation. Add 5 drops of a 1.0 per cent pyridine solution of phenolphthalein indicator and titrate with standard 0.5 N sodium hydroxide to a pink endpoint permanent for at least 15 seconds.

(3) Calculations:

$$\frac{\text{grams sample} \times 200}{(B-A)N} = \text{average molecular weight}$$

90 A=ml. of N normal NaOH required for the sample

B=average ml. of N normal NaOH required for the control

WHAT WE CLAIM IS:—

95 1. A filter for tobacco smoke comprising synthetic filaments or fibres, activated carbon particles and a high molecular weight polyalkylene glycol (as hereinbefore defined).

100 2. A filter according to claim 1, comprising synthetic filaments which are at least in part cellulose acetate.

3. A filter according to claim 2, wherein

the cellulose acetate filaments each have a denier of from 3 to 6.

4. A filter according to any preceding claim wherein the activated carbon is coconut carbon.

5. A filter according to any preceding claim wherein the activated carbon particles have a U.S. standard mesh size of from 10 to 44.

6. A filter according to claim 5, wherein the size of the activated carbon particles is such that 95% are retained on a 30 U.S. standard mesh screen and 95% pass through a 12 U.S. standard mesh screen.

7. A filter according to any preceding claim, wherein the high molecular weight poly-

alkylene glycol is polyethylene glycol, polypropylene glycol or methoxypolyethylene glycol.

- 5 8. A filter according to any preceding claim which contains from 2 to 200 parts by weight of carbon particles, and from 2 to 25 parts by weight of a high molecular weight polyalkylene glycol per 100 parts by weight of synthetic filaments or fibres.

- 10 9. A filter for tobacco smoke according to claim 1, substantially as herein described.

- 10 10. A filter for tobacco smoke constructed and arranged substantially as described herein with reference to and as illustrated in the
15 accompanying drawings.

11. A smoking article comprising tobacco

and a filter as claimed in any one of claims 1—9.

12. A process for preparing a tobacco smoke filter as claimed in claim 1, which comprises 20 coating or partially coating synthetic filaments with a high molecular weight polyalkylene glycol (as hereinbefore defined), applying to the coated or partially coated synthetic filaments a coating of carbon particles, and 25 forming the filaments into the required shape within a wrapping material.

13. A process according to claim 12, substantially as hereinbefore described.

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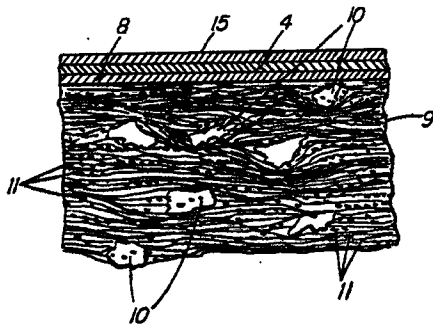
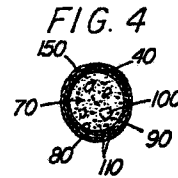
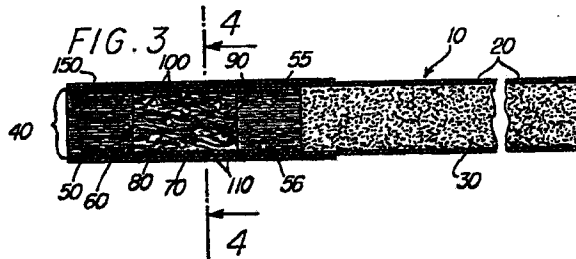
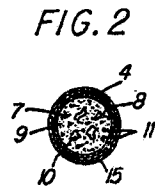
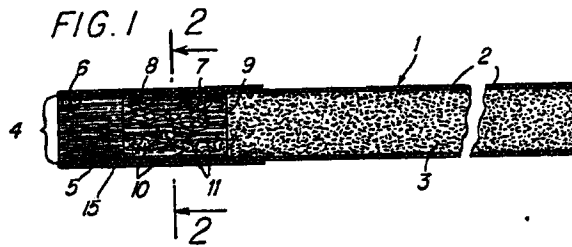


FIG. 5